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RESEARCH MEMORANDUM

PREPARATION AND PHYSICAL PROPERTIES

OF SOME TRIALKYLBORANES

By Louis Rosenblum and Harrison Allen, Jr.

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NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
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SUMMARY

Triethylborane, tri-n-propylborane, and tri-n-butylborane were prepared from boron trifluoride and the appropriate Grignard reagent. The first two compounds were distilled to purities of 99.8 and 99.7 mole percent, respectively, but tri-n-butylborane decomposed during distillation. The following physical properties of triethylborane and tri-n-propylborane were determined and compared with values from the literature: (1) freezing point, (2) heat of fusion, (3) boiling point, (4) heat of combustion, (5) refractive index, (6) density, (7) dielectric constant, and (8) infrared spectra. Experimental values for the refractive index of triethylborane, the heat of fusion of the tri-n-propylborane, and the dielectric constant of both compounds are reported here for the first time.

INTRODUCTION

The lower-molecular-weight trialkylboranes have heat-release properties intermediate between those of the boron hydrides and the conventional hydrocarbon fuels. In order to evaluate the potential of these organoboron compounds as high-energy fuels and as ignition sources, and to obtain fuel-system design information, it is necessary to have accurate values for several of their physical properties. A search of the literature revealed values for only some of these properties (refs. 1 to 6), and among the values were several large discrepancies. In order to obtain more accurate and consistent data, triethylborane, tri-n-propylborane, and tri-n-butylborane were synthesized and purified at the NACA Lewis laboratory.

There are several methods for the preparation of trialkylboranes reported in the literature (refs. 1 to 3 and 7 to 9). Most of the methods are variations of the general reaction:

$$BX_3 + 3\ RM \xrightarrow{\text{ether}} R_3B + 3\ MX$$

where

X = halide, or OR

R = alkyl group

M = MgX, 1/2 Hg, or 1/2 Cd

The reaction of boron trifluoride etherate with the appropriate Grignard reagent was reported to give good yields of the desired products (ref. 8) and was selected for the present work.

Triethylborane and tri-$\underline{n}$-propylborane were obtained in purities of 99.8 and 99.7 mole percent, respectively; tri-$\underline{n}$-butylborane was not obtained in a pure form since it decomposed during the distillation process.

The physical properties determined for the two purified compounds are (1) freezing point, (2) heat of fusion, (3) boiling point, (4) heat of combustion, (5) refractive index, (6) density, (7) dielectric constant, and (8) infrared spectra.

SYNTHESIS

General Procedure

The trialkylboranes were prepared by the following general reaction:

$$BF_3 \cdot R'_2O + 3\ RMgBr \xrightarrow{R_2O} R_3B + 3\ MgBrF$$

where

when R = ethyl, then R' = butyl

but when

R = butyl or propyl, then R' = ethyl

A low-boiling product such as triethylborane could be isolated by direct distillation from the reaction mixture (ref. 3). The higher-boiling trialkylboranes could not be isolated in this manner because of decomposition at the higher distillation temperature in the presence of copious amounts of salts. The salts and other hydrolyzable components can be removed by the addition of water upon completion of the reaction (ref. 8). Separation of the ether and aqueous layers and distillation of the ether yields the trialkylborane; however, the amount of product that can be prepared in this manner is limited by the practical size of the reaction vessel.

A modification of the existing procedure made possible the preparation of several batches of product without the usual dismantling, cleaning, and reassembling of equipment. When a little less than an equivalent amount of boron trifluoride ethyl etherate had been added to the Grignard reagent, there was a rapid separation of the mixture into two layers. The top layer consisted mainly of trialkylborane and ether, and the bottom layer consisted of excess Grignard reagent, salts and ether. The top layer could be siphoned from the reaction flask, more Grignard reagent added, and the operation repeated. Tri-n-propylborane and tri-n-butylborane were prepared by this modified procedure.

Materials

Ethyl, n-propyl, and n-butyl bromide from a commercial source were washed with water, dried over anhydrous sodium sulfate, and distilled. The refractive indices of the materials were n_d^{20} 1.4242, 1.4341, and 1.4399, respectively.

Boron trifluoride, 97-percent pure, was used as obtained from a commercial source.

Ethyl ether purchased commercially was dried over calcium hydride and was not purified further.

Butyl ether of commercial origin was percolated through a column packed with activated alumina, dried over calcium hydride, and used without further treatment.

EXPERIMENTAL DETAILS

The preparations of tri-n-butylborane and triethylborane are described in detail to illustrate the general procedure for the synthesis of the high- and low-boiling trialkylboranes, respectively.

Preparation

Tri-n-butylborane. - The n-butyl magnesium bromide was prepared in a 5-gallon stainless steel reactor by the addition of 20 moles of n-butyl bromide in 3 liters of ethyl ether to 20 moles of magnesium turnings in 2 liters of ethyl ether. The Grignard solution was stored in a 5-gallon tank under nitrogen until needed. An ether solution of boron trifluoride ethyl etherate was prepared by the slow addition with stirring of 6 moles of boron trifluoride to 2 liters of ethyl ether at 10° C.

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    The apparatus for the preparation of tri-n-butylborane consisted of
a graduated 12-liter three-neck flask equipped with a stirrer, a large-
capacity stainless steel condenser, and a graduated dropping funnel.
The entire apparatus was enclosed in a large dry box under a nitrogen
atmosphere.

    Three liters of Grignard solution were siphoned into the 12-liter
flask from the storage tank. Then boron trifluoride etherate solution
was added with stirring at such a rate (about 250 ml/hr) as to promote
vigorous refluxing. After approximately 650 milliliters of the boron
trifluoride etherate solution had been added, the reaction mixture sep-
arated quite suddenly into two layers, the bottom layer apparently con-
sisted of Grignard solution and magnesium salts. This layer, which was
black with white salt-like solids suspended throughout it reacted vigor-
ously with water, evolving gas. The clear pale-yellow top layer showed
no reaction with water and was apparently an ether solution of tri-n-
butylborane. This top layer was siphoned into a large separatory funnel
by means of a tube having a sintered-glass disk fused on one end to act
as a filter. When the entire top layer had been removed, another 3-liter
portion of Grignard reagent was added to the reaction flask, and the
entire procedure repeated. A total of 8300 milliliters of Grignard solu-
tion and 5.9 moles of boron trifluoride etherate was used. There was a
slight excess of Grignard reagent remaining after the last of the boron
trifluoride had reacted.

    The top layers obtained from the above reaction were washed in the
separatory funnel with a saturated aqueous solution of ammonium chloride
and then with water. Finally the ether solution was transferred to a
distillation flask and the ether removed by vacuum distillation. The
liquid residue which contained a small amount of salts was filtered. The
total weight of crude product was 989 grams; the yield was 91 percent
based on boron trifluoride.

    Triethylborane. - First ethyl magnesium bromide was prepared in a
5-gallon reactor by the addition of a solution of 48 moles of ethyl bromide
in 3 liters of butyl ether to a mixture of 48 moles of magnesium turnings
in 3 liters of butyl ether. The boron trifluoride butyl etherate was pre-
pared by the slow addition with stirring of 13 moles of boron trifluoride
to 4 liters of butyl ether at 10° C. This boron trifluoride solution was
added to the Grignard solution in the reactor over a period of $1\frac{1}{2}$ hours.

During the addition the outer jacket of the reactor was cooled so as to
maintain a reaction temperature of 40° to 45° C. When all the boron tri-
fluoride had been added, the reaction mixture was heated at 55° C for 2
hours. The triethylborane was then distilled out of the reactor directly
into a steel cylinder. Through the entire synthesis there was a positive
pressure of helium maintained in the reactor to exclude oxygen.

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Purification

The crude reaction products were each distilled through a 22-millimeter by 6-foot Podbielniak column. The entire distillation setup was enclosed in a dry box filled with a helium atmosphere (fig. 1).

The distillate was received and stored in 60-milliliter narrow-mouth, 19/22 standard taper, outside-ground bottles with 19/22 standard-taper hollow caps. A strong steel spring secured the cap to the bottle. This type of bottle permitted the use of silicone grease to aid in sealing while preventing contamination of the interior of the bottle (fig. 2).

Triethylborane was distilled at atmospheric pressure, while tri-n-propylborane and tri-n-butylborane were distilled at pressures of 20 and 10 millimeters of mercury, respectively. Triethylborane and tri-n-propylborane were successfully distilled, but tri-n-butylborane decomposed during distillation. The decomposition products adulterated the distillate besides causing the column to flood continuously. Lowering the pot temperature about 20° C by reducing the distillation pressure did not noticeably decrease the decomposition or the flooding. By rapidly distilling the compound through an 18-inch glass column, the amount of decomposition was reduced. However, the distillate still was not sufficiently pure to warrant determinations of physical properties.

Measurement of Physical Properties

Samples of triethylborane and tri-n-propylborane of 99.8 and 99.7 mole-percent purity, respectively, were used to obtain values (table I) for the following physical properties:

Freezing point. - The freezing curves were determined by means of a solenoid-stirred freezing-point apparatus (fig. 3) charged with 10-milliliter samples. The apparatus was loaded and sealed with a stopper in a dry box inerted with helium; subsequent operations were conducted outside the box. The thermometric system consisted of a 25-ohm platinum resistance thermometer, a resistance bridge (Müeller type, L&N #8069), and a highly sensitive galvanometer. The freezing point and the freezing point at zero impurity were obtained by analysis of the freezing curves (refs. 10 and 11). These data together with the values of the heat of fusion were used to calculate the purity of the samples (ref. 11). Duplicate determinations of the freezing point on a given sample differed by not more than 0.002° C.

Heat of fusion. - The heats of fusion were determined in a calibrated calorimeter similar to the one described in reference 12. The calorimeter was loaded by using a 25-milliliter pipette to meter out the sample. All operations were carried out in a dry box inerted with helium. A low-temperature slush bath was used as a uniform heat source to melt the

frozen sample, rather than the thermostated air bath used in reference 12. Analysis of the time-temperature curves of the sample and the heat source yielded a value for the heat of fusion. The estimated accuracy of the heat of fusion values was ± 0.2 calorie per gram.

Boiling point. - The boiling points were determined in an ebulliometer (ref. 13) equipped with a platinum resistance thermometer. The measurements were made in a helium atmosphere with an estimated accuracy of $\pm 0.1^{\circ}$ C.

Heat of combustion. - Heats of combustion were measured in a Parr adiabatic calorimeter with an Illium constant-volume bomb (ref. 14). The calorimeter was modified by using an electric heater to control the jacket water temperature. This heater was controlled by a thermoswitch which was activated by temperature differences between the bucket and the jacket. With this device it was possible to keep the average temperature variation between the bucket and jacket to $\pm 0.05^{\circ}$ C over the entire run. Even at the beginning of a run when temperature balance is difficult, the maximum deviation observed was only $\pm 0.1^{\circ}$ C.

Temperatures were measured with a platinum resistance thermometer which could be read to $\pm 0.001^{\circ}$ C. The bomb was calibrated by means of standard benzoic acid supplied by the Parr Instrument Company. Because of their volatility and susceptibility to oxidation, the samples of trialkylboranes were introduced into the bomb in small glass bulbs. The combustion procedure was the same as that used in reference 15. Completeness of combustion was determined by an analysis of the combustion products, carbon dioxide and boric acid. In general, combustion was 97 to 99 percent complete. Corrections were made for the unburned material on the assumption that it was exclusively elemental amorphous carbon and boron. Additional corrections were made to fit the combustion to the over-all process at 25° C:

$$BCH_{(liq.)} + O_2(gas, 1\ atm) \rightarrow$$

$$CO_2(gas, 1\ atm) + H_2O(gas, 1\ atm) + B_2O_3(amorphous)$$

The corrections made for incompleteness of combustion were satisfactory provided the combustion was at least 97 percent complete. In correcting for unburned carbon and boron, the uncertainty σ_n is approximately 0.2 percent. The uncertainty associated with the over-all raw heat of combustion σ_c is 0.3 percent. For a series of four heat-of-combustion determinations for triethylborane and for tri-n-propylborane, the standard deviation σ is 0.7 and 0.5 percent, respectively. For these determinations where $\sigma \simeq \sigma_n + \sigma_c$, the corrections appear to be good. When values for the heat of combustion for runs with less than 97 percent combustion are included in the data, the standard deviation

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increases so that $\sigma \gg \sigma_n + \sigma_c$. This indicates that for combustion less than 97 percent complete the assumption that elemental amorphous carbon and boron were the only unburned material present was not valid.

Refractive index. - The refractive indices were measured with a Bausch and Lomb precision Abbé refractometer enclosed in a dry box with a helium atmosphere (fig. 4). Measurements were made at $20.0^\circ \pm 0.1^\circ$ C and $30.0^\circ \pm 0.1^\circ$ C and three wave lengths of light at each temperature. The spectral lines used were the sodium D (5893A), the mercury Hg_g (5460.7A), and the mercury $Hg_{b.v.}$ (4358.3A). The estimated accuracy of the measurements was ± 0.0001. From the values for the refractive index and density, the molar refraction was calculated by means of the Lorentz-Lorenz equation.

Density. - The densities were measured with a modified Lipkin pycnometer. The modification consisted of fusing a standard taper male joint onto the ends of the pycnometer (fig. 5). These ends could then be capped with a female joint equipped with a small stopcock. Once the pycnometer was filled it could be sealed off without disturbing the level of the sample in the arms. The pycnometer was loaded and sealed in a dry box inerted with helium. The densities were measured outside the box at $20.00^\circ \pm 0.05^\circ$ C and $30.00^\circ \pm 0.05^\circ$ C with an accuracy of ± 0.0002 gram per milliliter.

Dielectric constant. - The dielectric constants were measured in a cell of 25-milliliter capacity (fig. 6 and ref. 16). After being filled in the dry box, the cell was sealed off and removed from the box for the capacity measurements. Measurements of the dielectric constant were made at $20.0^\circ \pm 0.1^\circ$ C and $30.0^\circ \pm 0.1^\circ$ C with a probable accuracy of ± 0.2 percent. The dielectric-constant apparatus has been previously described (ref. 17).

Infrared spectra. - The infrared spectra were obtained with a Baird doublebeam recording spectrophotometer. The precision of the instrument is ± 1 percent of the transmission value and ± 0.02 micron for the wave length. Undiluted samples were examined in a 0.06-millimeter sodium chloride cell and samples diluted with carbon tetrachloride in a 0.10-millimeter cell (fig. 7).

RESULTS AND DISCUSSION

An examination of the values listed in table I as compared with previous literature values (footnotes a to j in table I) shows discrepancies that cannot be explained in terms of experimental error. The cause of these discrepancies becomes evident when the trend in these values is examined. The refractive index reported for tri-n-propylborane (ref. 6) is much lower and the densities for triethylborane and tri-n-propylborane (refs. 2 and 6) are higher than the values obtained in the

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present investigation. When trialkylboranes are oxidized, the product
is alkyldialkoxyborane, $(RO)_2BR$, (ref. 18). Although no refractive index
and density data are available for this class of compounds, there are
data for the closely related trialkoxyboranes, $(RO)_3B$. The trialkyl-
boranes have higher refractive indices and lower densities than the cor-
responding trialkoxyboranes (refs. 6, 19, and 20). The alkyldialkoxy-
boranes would be expected to have values between those of the trialkyl-
boranes and the trialkoxyboranes. Therefore, contamination of the tri-
alkyboranes by oxidation products would lower the refractive index and
elevate the density values beyond those of the pure compound.

This explanation was further substantiated by an experiment run on
a sample of tri-n-propylborane. The sample was placed in the refractom-
eter and exposed to air. The refractive index over a period of several
seconds was observed to decrease rapidly as the sample was oxidized.

CONCLUDING REMARKS

Some of the characteristics of the trialkylboranes prepared in this
present investigation can now be examined in relation to their use as
fuels in an aircraft or missile.

As would be expected because of the high ratio of hydrocarbon con-
stituent to boron, the physical properties of these compounds are in gen-
eral very close to those of the analogous hydrocarbons. In comparing
JP-4 fuel with triethylborane and tri-n-propylborane, the densities of
the boranes were found to be about 10 percent lower and the heats of
combustion about 10 percent higher. Examination of the freezing and
boiling points of these compounds indicates that their liquid range is
adequate. The fact that dielectric properties of the trialkylboranes are
very similar to those of hydrocarbon compounds should permit use of the
boranes with the same type of capacitance gages now used to meter hydro-
carbon fuels.

The availability of these trialkylboranes at the present time is
poor, since they can be prepared only in laboratory quantities using
Grignard reactions. However, for large-scale preparation other procedures
may prove to be feasible, such as the reaction of diborane with an olefin
(ref. 21) or the reaction of an alkyl halide with boron trifluoride in the
presence of aluminum (ref. 9).

From the point of view of safety in handling these compounds, it
must be noted that their reactivity with air makes them hazardous. In
addition, they are most probably toxic. Individuals exposed to small
amounts of these trialkylboranes at the Lewis laboratory experienced head-
aches and nausea.

The most promising aspect of the lower-molecular-weight trialkyl-
boranes, besides their heats of combustion, is their ease of self-ignition.
Liquid triethylborane at room temperature will immediately ignite spon-
taneously when exposed to air. Even at partial pressures less than 1

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millimeter of mercury at 0° C, self-ignition occurs in oxygen (ref. 22).
This self-ignition phenomenon of triethylborane suggests that it could
be used as a high-energy ignition source in combustors. For higher-
molecular-weight boranes the spontaneous ignition delay increases, so
that when liquid tri-n-butylborane is exposed to air there is an induc-
tion period of several seconds before ignition occurs. Nevertheless,
their high reactivity suggests that these compounds may serve as fuels
for a stable pilot flame under severe operating conditions.

The alkylboranes, then, have physical characteristics similar to
those of hydrocarbons, but reactivities with air that are much superior.

Lewis Flight Propulsion Laboratory
    National Advisory Committee for Aeronautics
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TABLE I. - PHYSICAL PROPERTIES OF TRIETHYLBORANE AND TRI-N-PROPYLBORANE

	Freezing point, °C	Heat of fusion, kcal/mole	Boiling point (at 760 mm Hg), °C	Heat of combustion, Btu/lb	Temperature, t, °C	Refractive index, n			Molar refraction, R_D(5893A)	Density, d_4^t, g/ml	Dielectric constant
						n_{D}^{t}(5893A)	$n_{Hg_g}^{t}$(5460.7A)	$n_{Hg_{b.v.}}^{t}$(4358.3A)			
Tri-ethyl-borane	[a]-92.93	[b]3.3	[c]95.0	[d]20,670	20	1.3971	1.3988	1.4060	34.46	[e]0.6850	1.974
					30	1.3920	1.3939	1.4013		[e]0.6761	1.962
Tri-n-propyl-borane	[f]-64.92	2.6	[g]159.6	20,110	20	[h]1.4143	1.4164	1.4241	[i]48.43	[j]0.7232	2.026
					30	[h]1.4099	1.4119	1.4195		[j]0.7158	2.013

[a]-92.9° C (ref. 2).

[b]2.4 kcal/mole (ref. 23).

[c]95° C (ref. 2).

[d]20,160 ±0.4% Btu/lb (data from W. H. Johnson and E. J. Prosen of National Bureau of Standards)

[e]d_4^{23}, 0.6931 (ref. 2); d_4^{30}, 0.6774 (ref. 4).

[f]-65.5° C (ref. 5).

[g]156° C (ref. 3); 164.5° C (ref. 5).

[h]n_d^{20}, 1.4090 (ref. 6); $n_d^{22.5}$, 1.4135 (ref. 3).

[i]45.33 (ref. 6).

[j]d_4^{20}, 0.7639 (ref. 6); $d_4^{24.7}$, 0.7204 (ref. 3).

Figure 1. - Distillation apparatus for air- and moisture-sensitive materials.
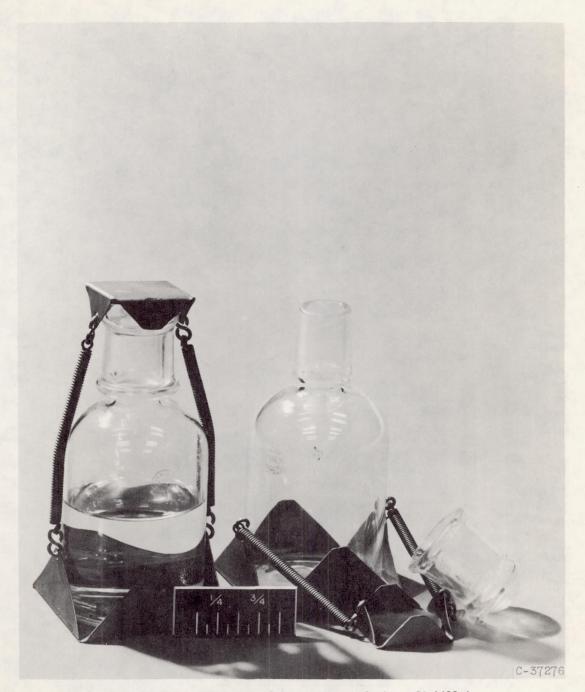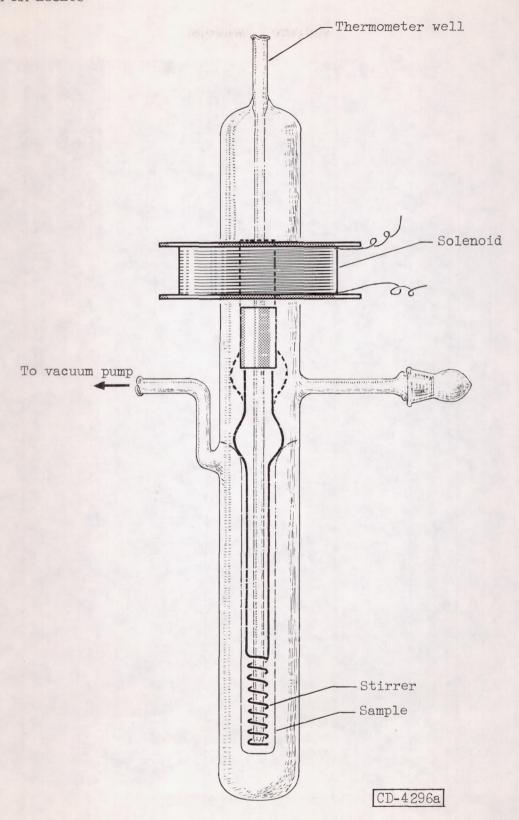
Figure 2. - Bottles used to receive and store distillate.
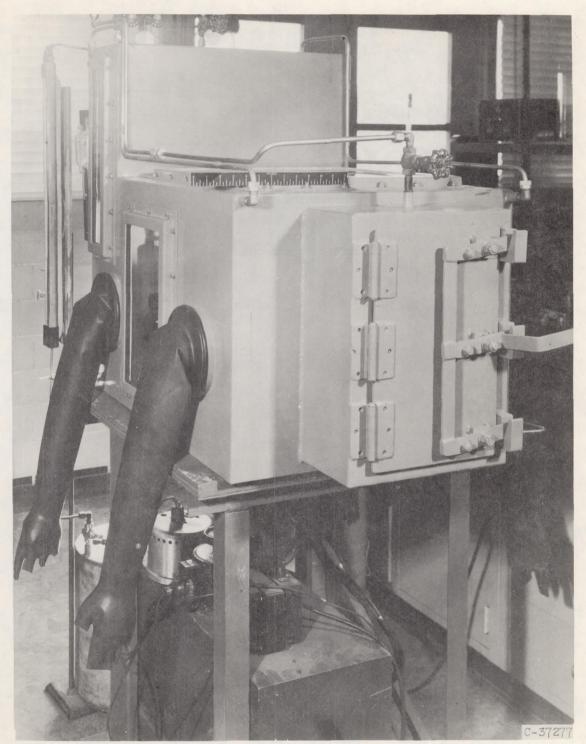
Figure 3. - Freezing-point apparatus.

Figure 4. - Physical-constants dry box.

CD-4296b
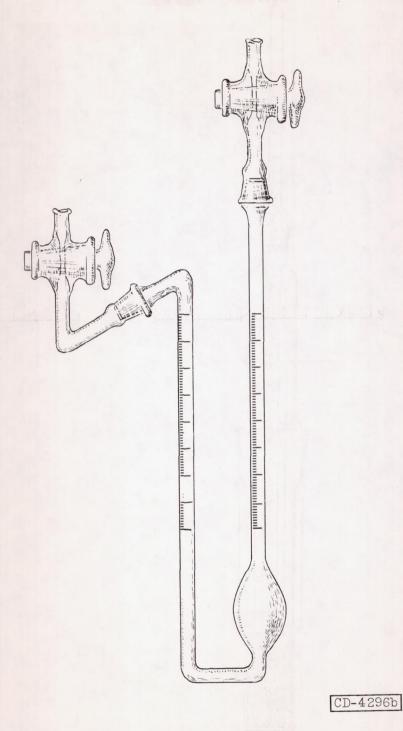
Figure 5. - Modified Lipkin pycnometer.

— Thermometer
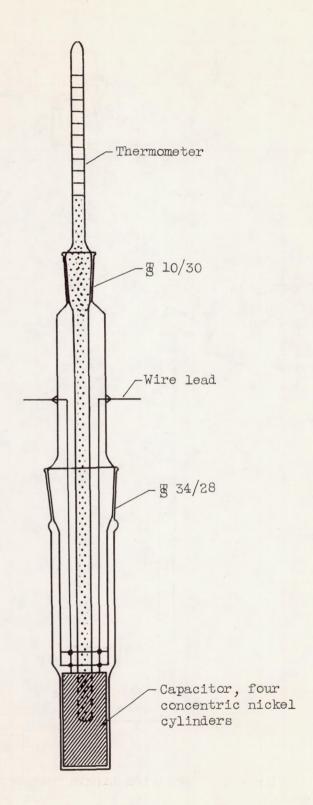
$\mathbb{F}$ 10/30

— Wire lead
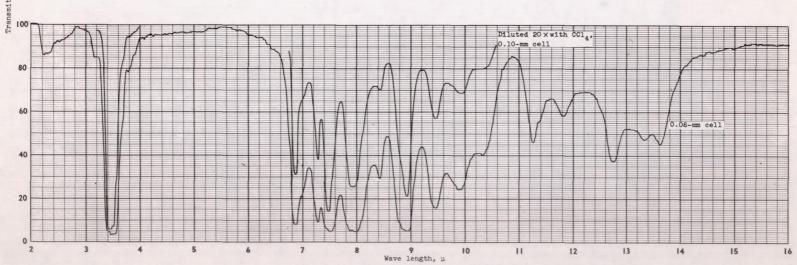
$\mathbb{F}$ 34/28

— Capacitor, four
concentric nickel
cylinders
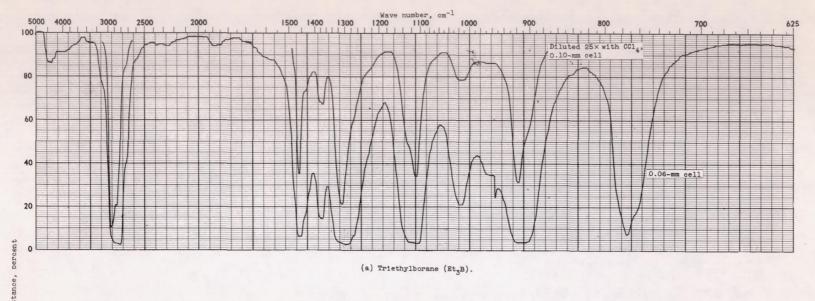
Figure 6. - Cell used to measure dielectric constants.

(a) Triethylborane (Et$_3$B).

(b) Tri-n-propylborane (n-Pr$_3$B).

Figure 7. - Infrared spectra.